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Ultrastrong Carbon Thin Films from Diamond to Graphene under Extreme Conditions: Probing Atomic-Scale Interfacial Mechanisms to Achieve Ultralow Friction and Wear

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14. ABSTRACT The aim of this research is to gain fundamental understanding of how to achieve low friction and wear in ultrastrong carbon-based materials. Experimentally, we use in situ nanotribometry that enables nanoscale visualization and quantification of atomic-level processes of sliding contacts inside the transmission electron microscope (TEM). These experiments are in turn modelled computationally using molecular dynamics, allowing better understanding of the atomic-scale processes controlling friction and wear. In this study, the interfacial behaviors (including adhesion forces and tribological performances) of diamond-like-carbon (DLC) and have been investigated. We find very consistent pull-in forces, indicating that adhesion before contact is dominated by van der Waals interactions. In contrast, large scatter in the pull-off forces is observed. However, they correlate to the average normal force applied during contact, suggesting atomic bonding is occurring at the interface. Construction of DLC computational models has begun, which aims to understand the atomistic mechanisms controlling the pull-in and pull-off. Our goal is to characterize and understand the atomic-scale mechanisms governing the tribological behavior of hard carbon materials during initial sliding contact, in order to understand what controls and enables the transition from high to low friction and wear. Developing this scientific insight is important to enable widespread usage of these materials to applications such as vibrating joints, contacting and sliding surfaces in micro- and nanoelectromechanical systems for sensors and actuators, joints in vacuum or anhydrous environments (as found in the upper atmosphere and in outer space), and protective coatings for bearings, bushings, and gears in engines, turbines and other aerospace components.					
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**Ultrastrong Carbon Thin Films from Diamond to Graphene under
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Abstract: We aim to gain a fundamental understanding of how to achieve low friction and wear in ultrastrong carbon-based materials. Experimentally, we use an *in situ* nanotribometry method, which enables nanoscale visualization of sliding contacts inside the transmission electron microscope (TEM). These experiments are in turn modelled computationally using molecular dynamics, allowing better understanding of the atomic-scale processes controlling friction and wear. In this study, the interfacial behaviors (including adhesion forces and tribological performances) of diamond-like-carbon (DLC) and have been investigated. Experimentally, we find very consistent pull-in forces, indicating that adhesion before contact is dominated by van der Waals interactions. In contrast, large scatter in the pull-off forces is observed. However, they correlate to the average normal force applied during contact, suggesting atomic bonding is occurring at the interface. Construction of DLC computational models has begun, which aims to understand the atomistic mechanisms controlling the pull-in and pull-off. The theoretical and experimental works are being prepared for publication. Our goal is to characterize and understand the atomic-scale mechanisms governing the tribological behavior (friction and wear) of hard carbon materials during initial sliding contact, in order to understand what controls and enables the transition from high to low friction and wear. Developing this scientific insight is important to enable widespread usage of these materials to applications such as vibrating joints^{1,2}, contacting and sliding surfaces in micro- and nanoelectromechanical systems for sensors and actuators³, joints in vacuum or anhydrous environments (as found in the upper atmosphere and in outer space), and

protective coatings for bearings, bushings, and gears in engines^{4,5}, turbines^{6,7}, and other aerospace components. The research was carried out using a unique *in situ* transmission electron microscopy (TEM) nanotribometry method that both PI's have collaboratively developed, thanks in part to prior AOARD support. This method allows us to visualize and quantify atomic-level processes occurring in sliding contacts through real time, real space, high resolution TEM measurements of a contact pair, where contact forces are precisely measured and controlled (More details in Section 2.1 and Figure 1).

1.1 Current Challenges in Tribology of Carbon-Based Materials

The mechanical and tribological properties of carbon-based films are, under the right conditions, superior to almost all other materials. Diamond, the stiffest and hardest bulk material known, can be grown in a smooth nanocrystalline thin film form with equivalent mechanical performance (ultranano-crystalline diamond - UNCD). These films can exhibit friction coefficients of 0.01 and less (as slippery as ice), and wear rates corresponding to fractions of an atomic layer per pass of the sliding interface ($<10^{-10} \text{ mm}^3 \text{N}^{-1} \text{m}^{-1}$) with no lubricant needed⁸⁻¹⁰. These characteristics far exceed those of well-lubricated interfaces of high performance steels and other expensive coatings.

Despite this potential, the widespread use of these materials is inhibited by a lack of scientific understanding of the initial process of wear, the so-called “run-in” process. While the steady-state wear rate and friction is often low, the *initial* rate of friction and wear during the “run-in” period, can be high. This “run-in” period may consist of only a few cycles of sliding, during which the contacting asperities become smoother, and local stresses, friction, and wear decrease, sometimes by *orders of magnitude*. However, in some cases, this “run-in” continues indefinitely¹¹⁻¹⁴. This is a catastrophic outcome: sliding parts can wear out far earlier than expected, or seize. Small changes in load, environment, sliding rate, surface roughness, and sample preparation have been observed to affect whether or not this crucial transition takes place. Results are largely empirical; there is a lack of scientific understanding of the atomic-scale processes in the contact that control whether the transition to low friction and wear occurs. Developing fundamental knowledge of these processes is crucial, and is the key to rationally designing and controlling coatings and operating parameters so that stable, low, reliable friction and wear performance can be obtained for engineered systems using these materials.

Previous studies by the PIs on interfacial contact conditions including atomic and macro scale studies show that the behavior of individual asperity contacts at the nanoscale plays a critical role in the run-in process¹⁵⁻¹⁹. More recent research, including by the both PI's, points toward specific atomic-scale mechanisms strongly affecting the sliding behavior of these materials, including: rehybridization from sp^3 to sp^2 -bonding of the C atoms²⁰, formation of bonds across the interface and subsequent atomic bond breaking²¹, and the strong passivating influence of small quantities of adsorbates^{9,22}. However, many of these observations are indirect or inferred, and others have not been studied in a systematic way that allows understanding of the run-in transition (namely the contact interactions on the surface separation and the real contact area for an asperity) to be investigated.

2 Experimental methodology

2.1 *In situ* TEM Tribometry

To address our research goals, over the last few years we have developed a unique *in situ* TEM tribometry method that allows atomic-scale characterization of a single-asperity sliding

contact. Both PI's Carpick and Jeng have a Hysitron PI-95 PicoIndenter TEM specimen holder, which was used for this purpose. This instrument measures normal load and displacement simultaneously inside a TEM, and can position an indenter in three dimensions with nanometer scale precision (Figure 1). While the standard configuration is designed for indentation, we developed a method for laterally sliding the indenter (by using integrated piezoelectric controls) against an atomic force microscope (AFM) cantilever. In this configuration, we can study the sliding behavior of several contact pairs (one material in the indenter, other material in the AFM tip) while observing the process with the TEM, allowing atomic-scale resolution of the mechanisms of deformation, adhesion, friction, wear, and failure at the sliding interface.

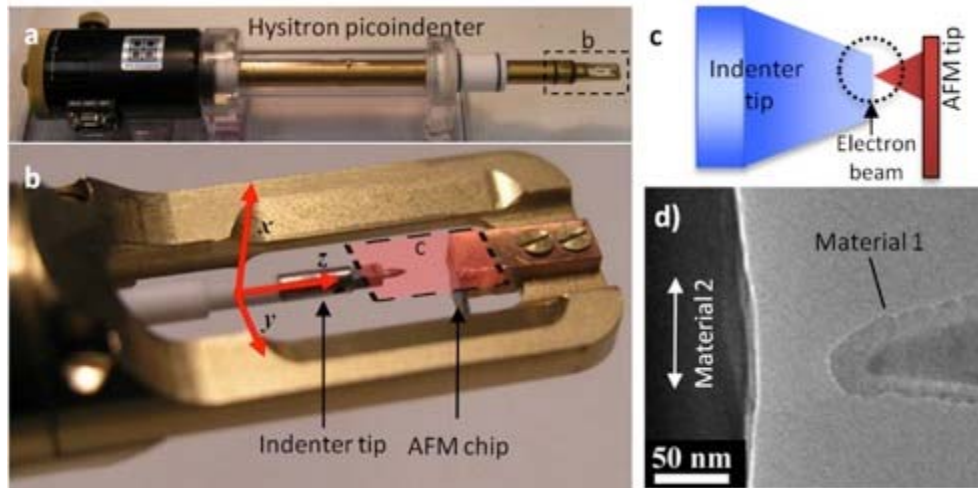


Figure 1: *In situ* TEM setup. (a) The Picoindenter TEM holder. The dashed rectangle is shown in b) in more detail. A piezo tube allows the indenter to attain three dimensional motion with sub-nm resolution. (c) Schematic close up of the AFM cantilever and indenter tip. (d) TEM micrograph of a typical experiment, where characterization of the sliding contact between the material of the AFM tip (#1) and the material of the indenter (#2) can be visualized with high resolution

The *in situ* TEM methodology has the key advantages of live-imaging the process of wear and of resolving volumes removed from the sliding asperity as low as 25 nm³ in volume²³. These capabilities are critical for probing the run-in period of wear. Moreover, since the measurements are carried out *in situ*, the analytical and crystal-characterization capabilities of the TEM can be leveraged to obtain further insights on the wear processes. All these can be done in sequence with sliding experiments to observe and understand the structural changes occurring.

As part of the present project, the PI developed and published a method for enabling characterization of AFM cantilever probes in the AFM²⁴. While scanning probe microscopy (SPM) enables detailed characterization of the geometric, mechanical, and transport properties of surfaces, the technique is often hindered by incomplete knowledge of the shape of the scanning probe. Various authors have measured tip geometry using scanning of known features or imaging of the tip using electron microscopy. The former technique requires complex numerical algorithms with multiple analysis parameters; the latter typically requires custom fixturing and suffers concerns about repeatability and contamination. Here we demonstrate a novel fixture and a practical guide to imaging SPM probes using electron microscopy, which minimizes or eliminates these disadvantages. The dissemination of this fixture and technique will enable a broader community of researchers to improve the quality of SPM imaging and the quantitative analysis of results.

The experimental procedure consists of bringing the indenter surface and AFM tip in

contact; the force is controlled by displacing the indenter beyond this point a given amount against the AFM tip, whose spring constant has been calibrated before. Subsequently, lateral sliding motion at a predetermined speed and duration is imposed on the contact. After sliding is finished, the contact is separated. This process is captured on video, allowing post-processing analysis for metrology of the force and observation of failure events. Furthermore, before and after sliding, high-resolution images of the tip are recorded, thus allowing visualization of volume loss (wear) due to sliding (quantified in post-processing), and of any structural change.

3 Results and Discussion

3.1 Adhesion of Diamond-like-carbon (DLC)

Diamond-like-carbon (DLC) is a promising material for a number of tribological applications such as hard-disk and machine-tool coatings, and aerospace applications. Understanding the behavior of the material in vacuum conditions is of particular importance for the latter applications. We performed adhesion studies of single-asperities made of DLC against single crystal diamond, while visualizing the contact process in-situ the TEM. Visualization of the contact allows to make direct correlations between the asperity's geometry and the adhesion measurements, also preventing difficulties of interpretation common in typical atomic force microscopy (AFM) experiments. To complement the experiments, we also carry out simulations of the same system. For experiments, we find very consistent pull-in forces, indicating that adhesive forces before contact arise from van der Waals (vdW) interactions. In contrast, large scatter in the pull-off forces is observed. However, they correlate to the time-averaged normal force during contact, suggesting atomic bonding is happening at the interface during contact. These observations agree with the general picture of an activation barrier required for bonding between the materials at the interface, which is reduced by mechanical stress.

To carry out the experiment, Atomic Force Microscopy (AFM) tips coated with DLC are brought in and out of contact on repeated occasions against a single crystal diamond surface, using the setup discussed in section 2.1. These experiments are carried out at CCU, with support and analysis conducted by Penn.

Measurements of the pull-in and pull-off forces are obtained by tracking features of the AFM tip throughout the frames of a video taken during the contact process. Pull-in and pull-off forces characterize adhesion before, and after contact, respectively. We also gain knowledge of the state of the material and contact geometry in real time.

Figure 2 shows the results of three separate experiments. For all experiments, it is evident that the pull-in forces are tightly concentrated around values near 1 nN, while the pull-off forces vary widely. Both the forces vary in a random way, *i.e.*, they do not follow a systematic trend. Note also that the dimensions of the tips do now vary significantly.

The low variability of the pull-in adhesive forces, combined with the insignificant variation in tip geometry suggests that the pull-in forces are dominated by long-range attractive forces, most likely van der Waals (vdW) forces. To confirm this, we extract the Hamaker constant from measurements of the pull-in forces, by calculating the theoretical interaction force between an axisymmetric punch and a flat, a situation closely resembling the experiment. In particular, the AFM tip can be modeled as an axisymmetric punch, *i.e.* a solid of revolution along the z-axis with the equation:

$$z = \frac{r^N}{NR^{N-1}} \# [1]$$

where N is an exponent characterizing the shape (2 is a paraboloid, higher powers represent flatter tips, such as those in the experiments), r is the radial distance and R is a characteristic size similar to a radius (exactly the radius for $N=2$).

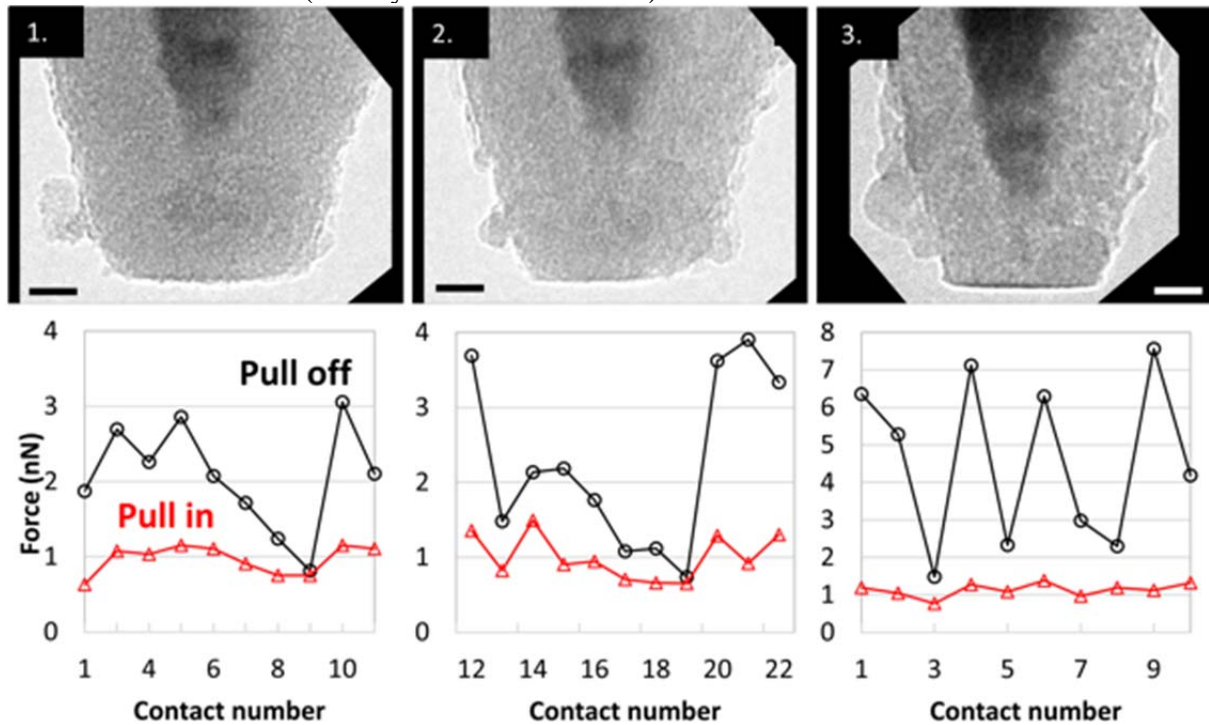


Figure 2: Results of three experiments for adhesion. Top: TEM images of the initial state of the tips. Scale bar: 10 nm. Bottom: Results for pull-in and pull-out forces are shown below their respective tip. Note that experiment 2 made use of the same tip as experiment 1, and so the contact number sequence is continued from the first experiment.

We have derived theoretical expressions for the vdW interaction energy between this solid and a flat. For brevity, we give only the final results, given by the following equation:

$$W(d) = -\frac{A_H \pi d^{-2+\frac{2}{N}} (-2+N) (NR^{-1+N})^{2/N}}{6 N^2 \sin\left(\frac{2\pi}{N}\right)}$$

where A_H is the Hamaker Constant, and d is the distance between the solid and the flat. The negative derivative of this expression with respect to distance is the force between solid and flat. Equating the derivative of this force with the cantilever spring constant (measured before the experiment) allows to solve for the Hamaker constant as a function of the measured pull-in force or distance as:

$$A_H = 6K \left(\frac{D_{pi}^{-4+\frac{2}{N}} \left(-3 + \frac{2}{N}\right) \left(-2 + \frac{2}{n}\right) (-2+N) \pi (NR^{-1+N})^{2/N}}{N^2 \sin\left(\frac{2\pi}{N}\right)} \right)^{-1}$$

To calculate the Hamaker constants, we fit the observed tip geometry to equation 1, and then use the measured pull-in distance. The average Hamaker constant obtained is $2.81 \pm 0.7 \times 10^{-19}$ J, very similar to that reported for the Hamaker constant for Diamond on Diamond under vacuum conditions (2.96×10^{-19} J)²⁵; the value for DLC on diamond is expected to

be similar and therefore is reasonable to conclude that the attractive pull-in observed is the result of van der Waals interactions. The pull-off on the other hand shows wide variations that cannot be explained by van der Waals adhesion alone. If this were the case, the forces would have a tight distribution, similar to that of the pull-in forces, but with a larger average. We are investigating the possibility that these forces vary due to the atomic bonding that happens after contact is established. Experimentally, this hypothesis is supported by the fact that pull-off forces are correlated to the time-averaged normal force between pull-in and pull-off i.e. when the surfaces are in contact, as shown in Figure 3.

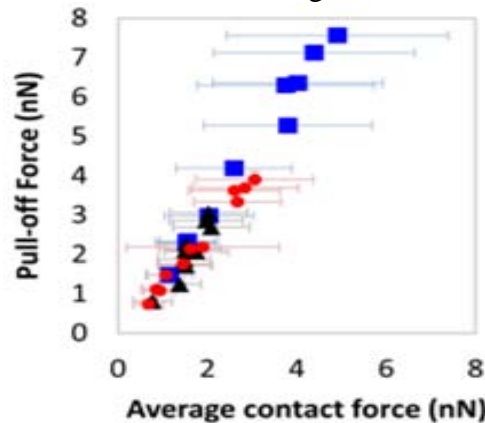


Figure 3: Experimental pull-off forces as a function of average contact force for the three experiments shown in Figure 2.

In the theoretical study, an atomic-scale model of mechanical contact behaviors between a DLC tip and a diamond (111) surface has been investigated by PI Prof. Jeng (CCU), in close resemblance to the experiments (Figure 2). During the initial approach of the DLC tip toward the diamond surface, an attraction (pull-in) occurs due to van der Waals forces between the DLC-diamond interfaces, where subsequently the end of a DLC tip forms a covalent bond across the interface. These bonds attached to the diamond surface are then broken as the DLC tip is retracted, leading to the DLC atoms remain on the diamond surface. The MD simulation results reveal that the bonding reactions such as bond formation and breaking across the interface plays a key role in wear mechanism at the micro/macroscale. Moreover, the results show that the number of DLC atoms adhered to the diamond plate increases as the downward displacement of a tip increases, particularly at lower strain rate.

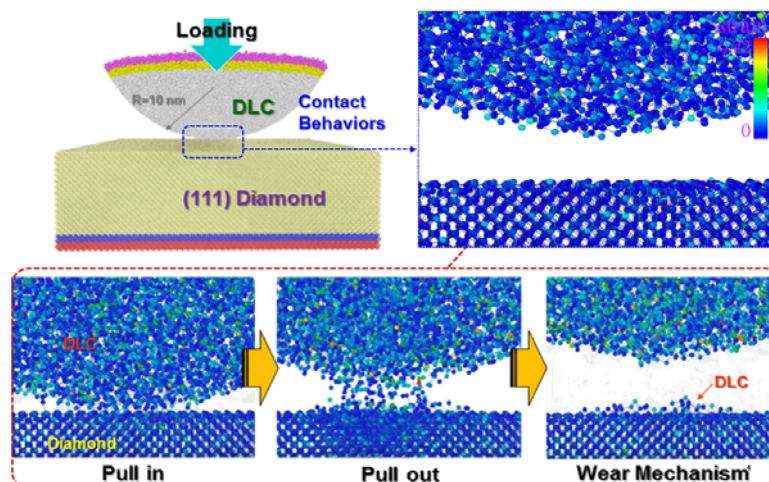


Figure 4: Atomic snapshots showing mechanical contact behavior of the DLC-diamond interface.

3.2 Study of DLC coatings: Hybrid DLC and carbon nanotube (CNT) composites

Minimizing the effects of inertial impacts and oscillating forces is a fundamental design problem in all dynamic mechanical systems, including those at the nano-scale³⁰⁻³². In systems characterized by reciprocating motion, effective energy absorbers are essential in improving their efficiency and prolonging their service lives. Recent advances in foamlike materials such as CNT-polymer composites³³⁻³⁵ and VACNT films³⁶⁻³⁸ provide an unprecedented opportunity to efficiently and reliably inject damping characteristics into load-bearing applications. DLC coatings can be used as a roof-layer to avoid the splitting failures of CNT films due to the impact response. In this study, hybrid carbon nanobuffers are developed by exploiting the ultra-hardness and wear-resistant properties of DLC coatings and the inherent viscoelasticity properties of vertically aligned CNT (VACNTs) (Figure 5).

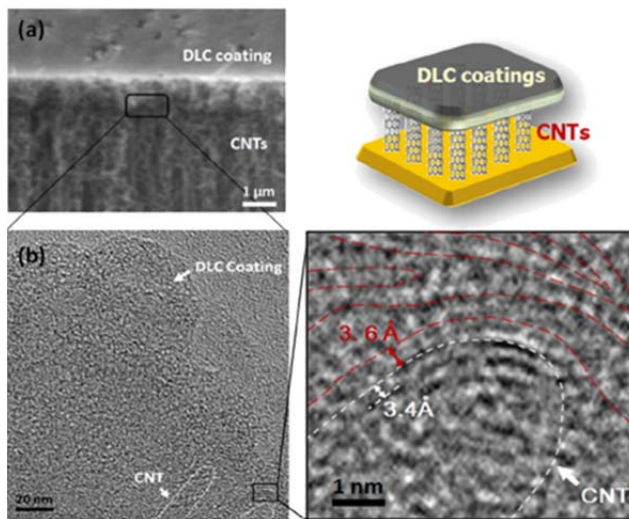


Figure 5: Cross-sectional SEM and TEM images of the DLC coating on top of the CNTs (region of HRTEM image showing the lattice spacing of intermediate graphitic shells between CNT and DLC coating is approximately 3.6 Å).

The viscoelastic properties of carbon nanobuffers incorporating thin-walled and thick-walled CNTs, respectively, are characterized by means of nanoindentation dynamic mechanical analysis tests. It is shown that the thin-walled nanobuffer has a better damping performance than the thick-walled nanobuffer due to its buckling-driven friction and post-buckling behaviors; particularly under large displacements. In addition, it is shown that under large indenter displacements, the VACNT arrays with DLC coatings display the improved stress distributions and enhanced strain energy dissipation performances due to the load transfer on the top of VACNTs (Figure 6).

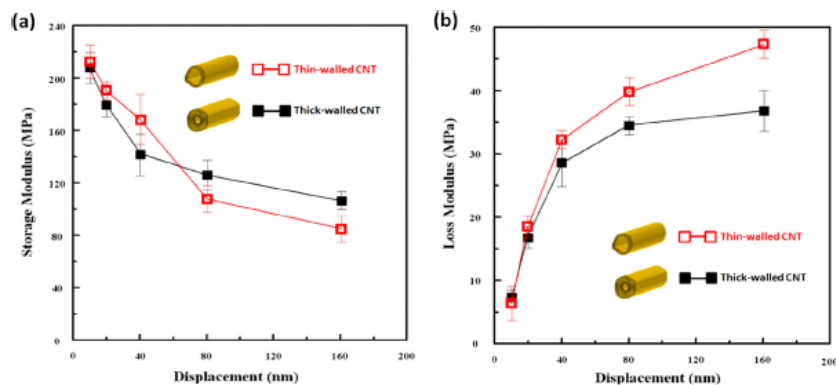


Figure 6 : (a) Storage and (b) loss moduli as function of loading displacement at constant loading frequency of 4 Hz for nanobuffer films containing thin- and thick-walled CNTs, respectively.

Molecular dynamics simulations are performed to investigate the roof-layer effect on damping behavior and structural deformation of the coated and uncoated VACNTs under nanoindentation. The results confirm that the VACNT with a DLC coating exhibits the significantly damping characterizations than the non-coated VACNT (Figure 7). Overall, the results presented in this study reveal the potential for tuning the damping performance of CNT-based nanobuffers through a careful control of the CNT size.

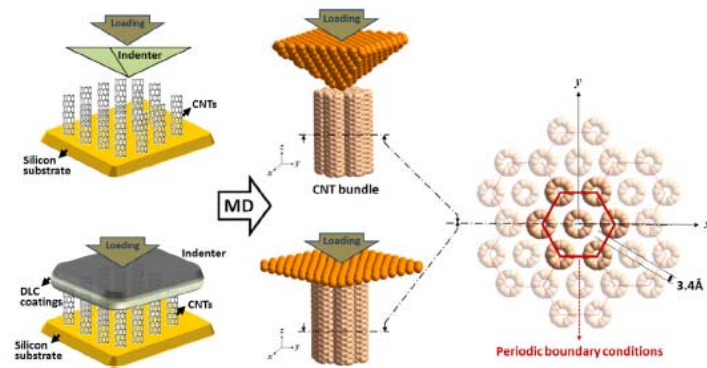


Figure 7: Schematic representation of MD simulation model for nanoindentation testing of CNT with and without DLC coating (Note that schematic cross-sectional diagram of a CNT bundle of six (5, 5) CNTs surrounding a core (5,5) CNT, where the nanotubes in the CNT bundle are spaced 3.4Å°)

3.3 Knowledge Transfer between the Collaborating Groups (Penn-CCU)

To strengthen collaboration between the two PI's and ensure exchange of best practices for carrying out experiments, PI Carpick visited CCU in August, 2016. During this visit, the PIs and project participants at CCU discussed and developed plans for experiments and joint publications. Pictures from the visit are shown in Figure 7.



Figure 7: Interactions between the PI's and project participants during Penn's personnel visit to Taiwan.

4 List of Publications and Significant Collaborations that resulted from AOARD support

4.1 Papers Published in Peer-Reviewed Journals

- P.C. Tsai, Y.R. Jeng, Effects of nanotube size and roof-layer coating on viscoelastic properties of hybrid diamond-like carbon and carbon nanotube composites, *Carbon* 86, 163-173 (2015).
- P.C. Tsai, Y.R. Jeng, In-situ nanomechanical study on bending characteristics of individual multi-walled carbon nanotubes, *Materials Science & Engineering A* 658, 389-392 (2016)

4.2 Papers Published in Non-Peer-Reviewed Journals or in Conference Proceedings

N/A

4.3 Conference Presentations

Invited

- Yeau-Ren Jeng, 2015, “In-situ Investigation of Lateral Junction Growth by Nanoindentation,” Symposium Keynote Speaker, 42nd International Conference On Metallurgical Coatings & Thin Films (ICMCTF), San Diego, USA, April 20 – 24
- Yeau-Ren Jeng, 2015, “A Journey from In-situ Characterizations of Nanomaterials to Industrial Applications,” Keynote Speaker, 19th Nano Engineering and Microsystem Technology Conference, Taipei, Taiwan, Aug. 13-14

4.4 Manuscripts Submitted but not yet Published

N/A

4.5 List of Interactions with Industry or with Air Force Research Laboratory (AFRL) scientists or significant collaborations that resulted from this work

- Industrial collaborations with Advanced Diamond Technologies Inc. (Gurnee, IL, USA), and Adama Innovations LLC (Ireland), on the development of diamond nanostructures for AFM probes.
- Collaborating with Hysitron Inc. (Minneapolis, MN, USA) on developing *in situ* tools.
- AFRL collaboration. Started with A. Voevodin (formerly at AFRL, now at U. North Texas) and C. Muratore, continuing with J. Bultman – AFRL and C. Muratore – U. Dayton/AFRL

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